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Part I [MTD 13: Ores and Raw Materials]



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*Indian Standard*

METHODS OF CHEMICAL  
ANALYSIS OF ILMENITE, PART I

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BUREAU OF INDIAN STANDARDS  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
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# Indian Standard

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# *Indian Standard*

## METHODS OF CHEMICAL ANALYSIS OF ILMENITE, PART I

### 0. FOREWORD

**0.1** This Indian Standard ( Part I ) was adopted by the Indian Standards Institution on 25 October 1972, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

**0.2** Ilmenite, an important ore of titanium, is used in the production of ferro-titanium and other industrial products. In order to determine correctly the chemical composition of ilmenite, need was felt to prescribe standard methods of chemical analysis, so that these may primarily serve as referee methods and may also be used by laboratories for routine analysis. Due consideration has been given in the preparation of this standard to the facilities available in the country for such analysis.

**0.3** The methods for determination of titania, ferrous oxide, chromium oxide, magnesium oxide, calcium oxide, vanadium oxide, etc, in Indian ilmenite ore are under investigation and will be covered in subsequent parts.

**0.4** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960\*.

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### 1. SCOPE

**1.1** This standard ( Part I ) covers methods for determination of silica, total iron as ferric oxide, zirconia, alumina, manganous oxide and phosphorus pentoxide in Indian ilmenite ores.

### 2. SAMPLING

**2.1** Samples shall be drawn and prepared in accordance with IS : 4166-1967†.

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\*Rules for rounding off numerical values ( revised ).

†Methods of sampling ilmenite and rutile.

### **3. QUALITY OF REAGENTS**

**3.1** Unless otherwise specified, 'pure chemicals' and distilled water ( *see* IS : 1070-1960\* ) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

### **4. GENERAL**

**4.1** In the methods prescribed in this standard, only relative number of Whatman filter papers have been prescribed. However, any other suitable brand of filter paper of corresponding porosity may be used.

### **5. DETERMINATION OF SILICA BY THE GRAVIMETRIC METHOD**

**5.1** The sample is fused with potassium pyrosulphate. The melt is extracted with dilute sulphuric acid, warmed and filtered. The residue after fusion with sodium carbonate is treated with dilute sulphuric acid and evaporated to fumes and filtered. Silica is determined from the residue as usual by the loss in weight after volatilization with hydrofluoric acid in presence of sulphuric acid.

#### **5.2 Reagents**

**5.2.1** *Potassium Pyrosulphate* — solid.

**5.2.2** *Dilute Sulphuric Acid* — 1 : 1, 1 : 9 and 3 : 97 ( *v/v* ).

**5.2.3** *Sodium Carbonate* — solid.

**5.2.4** *Hydrofluoric Acid* — 40 percent.

#### **5.3 Procedure**

**5.3.1** Transfer 0.5 g of an accurately weighed sample to a platinum crucible containing about 10 to 15 g of potassium pyrosulphate. Cover with a lid and heat at a low temperature till potassium pyrosulphate is melted. Increase the temperature to dull red heat. As soon as the sample is completely decomposed, remove the crucible from heat, and allow it to cool to room temperature.

**5.3.2** Transfer the crucible with its contents to a porcelain basin and extract the contents with 200 ml of dilute sulphuric acid ( 1 : 9 ). Warm the solution and filter through No. 40 filter paper. Remove any adhering particles from the basin by means of dilute sulphuric acid ( 3 : 97 ), wash

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\*Specification for water, distilled quality ( *revised* ).



the precipitate and filter paper for a few times with dilute sulphuric acid ( 3 : 97 ) and finally with hot water until acid free, collecting the filtrate and washing into a 500-ml volumetric flask.

**5.3.3** Transfer the filter paper alongwith the residue to a platinum crucible, ignite and fuse it with 2 grams of sodium carbonate. Extract the melt with about 25 ml of dilute sulphuric acid ( 1 : 1 ) in the basin and evaporate to dense white fumes. Continue fuming for 30 minutes. Cool and dilute to 50 ml with dilute sulphuric acid ( 1 : 9 ) and wash as under **5.3.2** and filter through No. 40 filter paper. Transfer the filtrate to the same 500-ml volumetric flask ( *see* **5.3.2** ).

**5.3.4** Ignite the filter along with the residue obtained under **5.3.3** in a platinum crucible at 1 050°C to constant weight. Moisten the residue with a few drops of dilute sulphuric acid ( 1 : 1 ) and add 5 to 10 ml of hydrofluoric acid. Evaporate to dryness, ignite, cool and weigh. Fuse this residue with one gram of potassium pyrosulphate and extract with 20 to 30 ml of dilute sulphuric acid ( 1 : 1 ).

**5.3.5** If the solution is clear, transfer it to the 500-ml volumetric flask ( *see* **5.3.2** ) and make up the volume to 500 ml. If there is any precipitate due to zirconium phosphate, then filter and wash. Fuse the precipitate with sodium carbonate. Digest the melt in 50 ml of hot water and filter. Wash with hot water, discard the filtrate. Dissolve the precipitate in hot dilute sulphuric acid ( 1 : 1 ) and add to the filtrate in the 500 ml volumetric flask ( *see* **5.3.2** ) and make up the volume to 500 ml. Preserve the solution for determination of total iron and zirconia, alumina and manganous oxide.

**5.3.6** Carry out a blank determination following the same procedure and using the same quantity of all the reagents but without the sample.

## 5.4 Calculation

$$\text{Silica, percent} = \frac{(A - B)}{C} \times 100$$

where

$A$  = mass in g of silica obtained under **5.3.4**,

$B$  = mass in g of silica obtained from the blank, and

$C$  = mass in g of the sample taken.

## 6. DETERMINATION OF TOTAL IRON AS FERRIC OXIDE BY THE VOLUMETRIC METHOD

**6.1 Outline of the Method** — Ferrous iron is first oxidized and the total ferric iron then reduced with stannous chloride, excess of which is oxidized by mercuric chloride. The reduced iron is titrated with standard potassium

## IS : 6744 ( Part I ) - 1972

dichromate<sup>\*</sup> solution using sodium diphenylamine sulphonate solution as indicator.

### 6.2 Reagents

**6.2.1 Concentrated Nitric Acid** — sp gr 1.42 ( conforming to IS : 264-1968\* ).

**6.2.2 Concentrated Ammonium Hydroxide** — sp gr 0.90.

**6.2.3 Ammonium Chloride Solution** — 10 percent ( *m/v* ).

**6.2.4 Dilute Hydrochloric Acid** — 1 : 1 ( *v/v* ).

**6.2.5 Concentrated Hydrochloric Acid** — sp gr 1.16 ( conforming to IS : 265-1962† ).

**6.2.6 Stannous Chloride Solution** — Dissolve by heating 60 g of pure stannous chloride in a mixture of 400 ml of concentrated hydrochloric acid and 600 ml of water until the solution is complete. Cool, add a few pieces of granulated tin and preserve the solution in an air-tight bottle.

**6.2.7 Mercuric Chloride Solution** — Prepare a saturated solution of mercuric chloride in water.

**6.2.8 Sulphuric Acid-Phosphoric Acid Mixture** — Add slowly 150 ml of concentrated sulphuric acid to 700 ml of water with continuous stirring. Add to this solution 150 ml of syrupy phosphoric acid. Destroy the oxidizable impurities by adding potassium permanganate solution ( 0.1 N ) drop by drop until the pink colour of permanganate persists. Cool the solution.

**6.2.9 Sodium Diphenylamine Sulphonate Indicator Solution** — Dissolve 0.2 g of the reagent in 100 ml of hot water.

**6.2.10 Standard Potassium Dichromate Solution** — 0.1 N. Dry pure potassium dichromate at 105 to 110°C for one and a half hours. Cool in a desiccator and transfer exactly 4.904 g to a one-litre volumetric flask carefully through a funnel using water at room temperature. Wash the funnel thoroughly and dissolve the reagents by shaking until the solution is complete. Make up to the mark and mix well.

### 6.3 Procedure

**6.3.1** Pipette out exactly 100 ml of the solution preserved in 5.3.5 into a 500-ml beaker. Add 5 ml of concentrated nitric acid and heat to expel brown fumes. Cool, neutralize first with concentrated ammonia and finally with dilute ammonia. Add dilute ammonium hydroxide ( 1 : 1 ) until slightly but distinctly alkaline. Boil for few minutes and allow to settle

\*Specification for nitric acid ( *first revision* ).

†Specification for hydrochloric acid ( *revised* ).

for some time. Filter while hot through No. 41 filter paper and wash for a few times alternately with ammonium chloride solution and hot water.

**6.3.2** Dissolve the precipitate through the filter paper with 20 ml of dilute hydrochloric acid and wash thoroughly with hot water collecting the filtrate and washings in a 500-ml conical flask. Rinse the beaker with little hot dilute hydrochloric acid and transfer it to the flask. Evaporate but do not boil to 40 ml and add 30 ml of concentrated hydrochloric acid. Reduce iron by adding stannous chloride solution dropwise to the hot solution until it becomes colourless. Add two to three drops of stannous chloride solution in excess. Cool the solution to room temperature.

**6.3.3** Add excess of mercuric chloride solution ( about 5 ml ) in one instalment and shake the flask. At this stage a silky white\* precipitate appears. Keep for about 5 minutes and add 15 ml of sulphuric-phosphoric acid mixture, and dilute to about 300 ml, washing down the sides of the flask. Add three to four drops of sodium diphenylamine sulphonate indicator solution and titrate slowly with standard potassium dichromate solution stirring continuously until the green colour begins to darken. Continue the titration slowly till a drop of the dichromate solution produces a stable violet-blue colouration.

**6.3.4** Make a blank determination following the same procedure and using the same amounts of all reagents but without the sample.

## 6.4 Calculation

$$\text{Total iron expressed as ferric oxide, percent} = \frac{(A - B) C \times 7.985}{D}$$

where

$A$  = volume in ml of the standard potassium dichromate solution required for the test solution,

$B$  = volume in ml of the standard potassium dichromate solution required for the blank,

$C$  = normality of the standard potassium dichromate solution, and

$D$  = mass in g of the sample represented by the aliquot taken.

## 7. DETERMINATION OF ZIRCONIA BY THE GRAVIMETRIC METHOD

**7.1 Outline of the Method** — Zirconium is precipitated as hydroxide along with other hydroxides and then the precipitates are treated with sodium hydroxide. The residue is dissolved and zirconium is precipitated from the filtrate as phosphate and weighed as  $\text{ZrP}_2\text{O}_7$ .

\*If it fails to appear or if a greyish precipitate appears, reject the test.

## 7.2 Reagents

- 7.2.1 Concentrated Nitric Acid** — sp gr 1.42 ( conforming to IS : 264-1968\* ).
- 7.2.2 Dilute Ammonium Hydroxide** — 1 : 1 ( *v/v* ).
- 7.2.3 Ammonium Chloride** — solid.
- 7.2.4 Methyl Red Indicator Solution** — 0.1 percent ( *m/v* ). Dissolve 0.1 g of methyl red in 3.7 ml of sodium hydroxide solution ( 0.1 N ) and dilute to 250 ml with water, filter if necessary.
- 7.2.5 Ammonium Nitrate Solution** — 5 percent ( *m/v* ).
- 7.2.6 Sodium Hydroxide** — solid.
- 7.2.7 Dilute Hydrochloric Acid** — 1 : 1 and 1 : 2 ( *v/v* ).
- 7.2.8 Dilute Sulphuric Acid** — 1 : 4 ( *v/v* ).
- 7.2.9 Hydrogen Peroxide** — 20 volumes.
- 7.2.10 Diammonium Hydrogen Phosphate** [ (  $NH_4$  )<sub>2</sub>  $HPO_4$  ] — solid.

## 7.3 Procedure

**7.3.1** Pipette out exactly 200 ml of the solution preserved under **5.3.5** into a 500-ml beaker, add 5 ml of concentrated nitric acid to oxidize ferrous iron and boil. Cool, and add 4 to 5 g of ammonium chloride and 5 drops of methyl red indicator. Precipitate hydroxides by adding first concentrated ammonium hydroxide and then with dilute ammonium hydroxide until the colour changes to the distinct yellow. Allow to settle, filter and wash four times with hot ammonium nitrate solution. Digest the precipitate of hydroxides with about 3 g of sodium hydroxide for an hour on steam-bath, dilute, filter and wash.

**7.3.2** Dissolve the precipitate in dilute hydrochloric acid ( 1 : 1 ) and repeat the digestion of the residue. Add the filtrate to the filtrate under **7.3.1**. Concentrate the solution and preserve it for the determination of alumina.

**7.3.3** Dissolve the precipitate in hot dilute hydrochloric acid ( 1 : 2 ) and add sufficient dilute sulphuric acid so that the solution contains 10 percent by volume of sulphuric acid. Add sufficient hydrogen peroxide to complex all the titanium ( an excess does no harm ) and 0.5 g of diammonium hydrogen phosphate, allow to stand overnight filter, and wash with ammonium nitrate solution till free from sulphate.

**7.3.4** Transfer the paper and the residue to a weighed platinum crucible and heat very carefully so that the paper does not burn. When the paper has charred, gradually increase the temperature until all carbon is removed

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\*Specification for nitric acid ( *first revision* ).

and then heat at about 1 050°C to constant weight. Cool in a desiccator and weigh as  $\text{ZrP}_2\text{O}_7$ .

#### 7.4 Calculation

$$\text{Zirconia, percent} = \frac{A \times 46.46}{B}$$

where

$A$  = mass in g of  $\text{ZrP}_2\text{O}_7$ , obtained under 7.3.4, and

$B$  = mass in g of the sample represented by the aliquot taken.

### 8. DETERMINATION OF ALUMINA BY THE OXINATE ( VOLUMETRIC ) METHOD

**8.1 Outline of the Method** — Aluminium is precipitated as oxinate from a buffered acetate solution and dissolved in hydrochloric acid followed by bromometric titration of the liberated 8-hydroxyquinoline.

#### 8.2 Reagents

**8.2.1 Concentrated Hydrochloric Acid** — sp gr 1.16 ( conforming to IS : 265-1962\* ).

**8.2.2 Hydroxylamine Hydrochloride Solution** — 100 g/l in water.

**8.2.3 o-Phenanthroline Solution** — Dissolve one gram of o-phenanthroline monohydrate in 90 ml of water with gentle heating and constant stirring. Cool and dilute to 100 ml.

**8.2.4 Bromocresol Purple Indicator Solution** — 0.4 g/l. Dissolve 0.04 gram of bromocresol purple indicator in 100 ml of hot water, cool and filter.

**8.2.5 Dilute Ammonium Hydroxide** — 1 : 3 ( v/v ).

**8.2.6 8-Hydroxyquinoline ' Oxine ' Solution** — Dissolve 5 g of 8-hydroxyquinoline in 15 ml of glacial acetic acid by warming and dilute with water to 250 ml.

**8.2.7 Ammonium Acetate** — solid.

**8.2.8 Potassium Bromide** — solid.

**8.2.9 Standard Potassium Bromate Solution** — 0.05 N. Dry some finely powdered ( AR ) potassium bromate for 1 to 2 hours at 120°C and allow to cool in a closed vessel in a desiccator. Weigh out accurately 1.392 g of the pure potassium bromate and dissolve it in one litre of water in a volumetric flask.

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\*Specification for hydrochloric acid ( revised ).

**8.2.10 Potassium Iodide Solution** — 10 percent ( *m/v* ).

**8.2.11 Standard Sodium Thiosulphate Solution** — 0.05 N. Weigh out 12.5 g of ( AR ) sodium thiosulphate crystals, dissolve in freshly boiled and cooled water and make up to one litre in a volumetric flask. If the solution is to be kept for more than a few days, add 0.1 g of sodium carbonate or three drops of chloroform. Standardize the solution against standard potassium bromate solution iodimetrically as in **8.3.4**.

**8.2.12 Starch Solution** — Make a suspension of 1 g of soluble starch in about 5 ml of water and add it carefully to 100 ml of boiling water. Cool the solution before use. Prepare fresh as needed.

**8.3 Procedure**

**8.3.1** Acidify the filtrate preserved for determination of alumina under **7.3.2**, with concentrated hydrochloric acid. Concentrate the solution to about 150 ml. Add 2 ml of hydroxylamine hydrochloride followed by 2 ml of *o*-phenanthroline solution. Cool, add three to four drops of bromocresol purple indicator solution and dilute with ammonium hydroxide until the colour of the solution just turns violet. Keep the solution on a hot-plate. When the solution attains the temperature of 60 to 70°C, add 25 to 30 ml of oxine solution and 7 to 8 g of ammonium acetate slowly with stirring. Maintain the solution at 60 to 70°C until the precipitate is coagulated. Filter through sintered glass crucible of porosity No. 4 and wash the precipitate with hot water 10 to 12 times.

**8.3.2** Dissolve the oxinate complex in minimum quantity of warm concentrated hydrochloric acid and collect the solution in a 500-ml conical flask. Wash the crucible with hot water till acid-free. Collect the washings also in the same conical flask.

**8.3.3** Cool the solution, add about 2 to 3 g of potassium bromide and shake till the solid dissolves. Now add exactly 20 ml of standard potassium bromate solution and keep for about 10 minutes for complete bromination. Add 10 ml of potassium iodide solution and dilute it to 200 ml. Titrate the liberated iodine with standard sodium thiosulphate solution using starch as indicator.

**8.3.4** Pipette out 20 ml of standard bromate solution separately into a 500-ml conical flask. Add 2 to 3 g of potassium bromide till solid dissolves. Add about 10 ml of potassium iodide solution followed by about 5 ml of concentrated hydrochloric acid. Dilute the contents to about 200 ml and titrate the liberated iodine against standard sodium thiosulphate solution using freshly prepared starch as indicator.

**8.4 Calculation**

$$\text{Alumina, percent} = \frac{(A - B) \times C \times 0.425}{D}$$

where

$A$  = volume in ml of the standard sodium thiosulphate solution required in 8.3.4,

$B$  = volume in ml of the standard sodium thiosulphate solution required for the unreacted bromate solution,

$C$  = normality of the standard sodium thiosulphate solution, and

$D$  = mass in g of the sample represented by the aliquot taken.

## 9. DETERMINATION OF MANGANOUS OXIDE BY THE PERIODATE ( PHOTOMETRIC ) METHOD

**9.1 Outline of the Method** — After removal of silica, manganese is oxidized to permanganic acid by means of potassium periodate and determined photometrically at 525 nm.

### 9.2 Reagents

**9.2.1 Phosphoric Acid** — 85 percent.

**9.2.2 Potassium Periodate Solution** — Cautiously add 25 ml of concentrated sulphuric acid to 50 ml of water. Cool and dilute to 100 ml. Dissolve 7.5 g of potassium periodate in this solution.

**9.2.3 Standard Manganese Stock Solution** ( 1 ml = 0.25 mg of Mn ) — Dissolve a weighed quantity of electrolytic pure manganese of known manganese content in 5 ml of dilute nitric acid ( 1 : 1 ), the quantity weighed being such as will give 0.250 0 g of manganese; cool and dilute to one litre.

### 9.3 Procedure

**9.3.1** Concentrate 100-ml aliquot from 5.3.5 in a 250-ml beaker to 50 ml, and add 5 ml of phosphoric acid and 10 ml of potassium periodate solution. Heat to boiling and boil gently for two minutes. Then digest just below the boiling point for 10 minutes to develop full intensity of the colour. Cool, transfer to a 100-ml volumetric flask, dilute to the mark and mix.

**9.3.2** Transfer a suitable portion of the blank to an absorption cell and adjust the photometer to initial setting using a light filter at approximately 525 nm. While maintaining this photometer adjustment, take photometric readings of an aliquot of the solution prepared under 9.3.1.

**NOTE** — If zirconium is present, the solution should be centrifused ( 25 ml of the turbid solution be centrifused for 3 minutes ) and suitable aliquot of the supernatant solution decanted and transferred to the absorption cell for photometric measurement.

**9.3.3 Calibration Curve** — Take a series of standard manganese solution 0, 1, 2, 3 and 4 ml and a reagent blank. Using same quantities of all

reagents carry through all the stages of procedure and record the photometric readings of all the standard solutions along with the blank. Draw a calibration curve by plotting the photometric readings of the standard solutions against mg of manganese per 100 ml of the solution.

**9.4 Calculation** — Convert the photometric readings of the sample to milligram of manganese by means of the calibration curve, and calculate the percentage of manganous oxide as follows:

$$\text{Manganous oxide, percent} = \frac{A}{B} \times 0.129$$

where

$A$  = mass in mg of manganese found in the aliquot after correction for the blank, and

$B$  = mass in g of the sample represented by the aliquot taken.

NOTE — Suitable correction for chromium may be needed when it is present in amount that will cause interference in this determination.

## 10. DETERMINATION OF PHOSPHORUS PENTOXIDE BY THE MOLYBDATE (ALKALIMETRIC) METHOD

**10.1 Outline of the Method** — The sample is fused with sodium peroxide and melt extracted in water. After separation of silica from the filtrate, phosphorus is co-precipitated with iron and finally phosphorus is precipitated as ammonium phosphomolybdate. The precipitate is filtered, washed and dissolved in excess of standard alkali. The unreacted alkali is back titrated against standard hydrochloric acid.

### 10.2 Reagents

**10.2.1 Sodium Peroxide** — solid.

**10.2.2 Sodium Hydroxide Solution** — 2 percent (  $m/v$  ).

**10.2.3 Dilute Hydrochloric Acid** — 1 : 1 (  $v/v$  ).

**10.2.4 Concentrated Hydrochloric Acid** — sp gr 1.16 ( conforming to IS : 265-1962\* ).

**10.2.5 Ferric Chloride Solution** — 10 percent (  $m/v$  ).

**10.2.6 Concentrated Ammonium Hydroxide** — sp gr 0.90.

**10.2.7 Concentrated Nitric Acid** — sp gr 1.42 ( conforming to IS : 264-1968† ).

\*Specification for hydrochloric acid ( revised ).

†Specification for nitric acid ( first revision ).



**10.2.8 Hydroxylamine Hydrochloride Solution** — 1.5 percent ( *m/v* ).

**10.2.9 Ammonium Nitrate** — solid.

**10.2.10 Ammonium Molybdate Solution** — Add solution *A* to solution *B* with constant stirring, then add 0.1 gram of ammonium phosphate dissolved in 10 ml of water and let it stand for at least 24 hours before using. Keep the solution in a cool place and always filter before using. Prepare solutions *A* and *B* as follows:

a) *Solution A* — Dissolve 100 g of molybdic acid ( 85 percent  $\text{MoO}_3$  ) in a mixture of 150 ml of concentrated ammonium hydroxide and 270 ml of water.

b) *Solution B* — Mix 537 ml of concentrated nitric acid and 1 280 ml of water and cool.

**10.2.11 Dilute Nitric Acid** — 1 : 99 ( *v/v* ).

**10.2.12 Potassium Nitrate Wash Solution** — one percent ( *m/v* ).

**10.2.13 Sodium Hydroxide Solution** — 0.1 N approximately ( carbonate-free ).

**10.2.14 Phenolphthalein Indicator Solution** — Dissolve 0.5 gram of the phenolphthalein in 100 ml of rectified spirit.

**10.2.15 Standard Hydrochloric Acid** — 0.1 N. Dilute about 11 ml of concentrated hydrochloric acid to one litre and standardize against sodium carbonate.

### 10.3 Procedure

**10.3.1** Weigh accurately one gram of the sample into an iron crucible and fuse with about 5 g of sodium peroxide. Extract the melt in about 100 ml of water in a 400-ml beaker. Add about 1 to 2 g of sodium peroxide to the water extract in the beaker, boil for about 5 minutes and filter. Wash the residue thoroughly with hot sodium hydroxide solution. Collect the filtrate and washings in the same beaker.

**10.3.2** Neutralize the above filtrate with dilute hydrochloric acid providing an excess of 20 ml of concentrated hydrochloric acid. Add 10 ml of ferric chloride solution. Set the whole solution for evaporation, drying and baking. Extract the baked mass in about 20 ml of concentrated hydrochloric acid, dilute, filter, and wash to make free from acid by means of hot water. Boil and bring down the volume of the filtrate and the washings to about 100 ml. Transfer the solution to a 500-ml conical flask.

**10.3.3** Neutralize the hot filtrate ( 80 to 100°C ) with concentrated ammonium hydroxide until a slight persistent precipitate is formed. Redissolve the precipitate in a few drops of concentrated nitric acid providing

finally about 2 ml of nitric acid in solution in excess. ( If the sample contains vanadium, add 2 ml of hydroxylamine hydrochloride solution. ) Add about 10 g of ammonium nitrate. Maintain the temperature of the solution between 40 to 50°C. Add about 40 ml of ammonium molybdate solution. Shake the flask vigorously for about 10 minutes and let it stand for about 30 to 45 minutes.

**10.3.4** Filter the precipitate through a thick pad of filter paper pulp prepared on a perforated disc in a funnel. Wash the flask and the precipitate two to three times with dilute nitric acid and then with potassium nitrate wash solution till 10 ml of the filtrate collected in a test-tube does not consume more than one drop of sodium hydroxide solution using a drop of phenolphthalein indicator solution ( testing the filtrate from third wash ). Transfer the pad of paper pulp with the precipitate back to the flask in which phosphorus was precipitated and wash down any precipitate adhering to the funnel with a jet of water, into the flask. Add 50 to 60 ml of water and add with stirring sodium hydroxide solution from a burette until all the yellow precipitate has dissolved and the solution is colourless. Add about 5 ml of sodium hydroxide solution in excess and stir. Add three to four drops of phenolphthalein indicator solution and titrate with standard hydrochloric acid until the pink colour is completely discharged.

**10.3.5** Carry out a blank determination on the same quantity of sodium hydroxide solution which was added in the test, and titrating with standard hydrochloric acid, using three to four drops of phenolphthalein indicator solution.

## 10.4 Calculation

$$\text{Phosphorus pentoxide, percent} = \frac{(A - B)C \times 0.309}{D}$$

where

$A$  = volume in ml of standard hydrochloric acid required for the blank test,

$B$  = volume in ml of standard hydrochloric acid required for the sample,

$C$  = normality of standard hydrochloric acid, and

$D$  = mass in g of the sample taken.

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